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Mendeleev Commun., 2011, **21**, 50–52
Mendeleev Communications

New approach to *N,N*-dialkoxy-*N′*-arylureas and *N,N*-dialkoxycarbamates†

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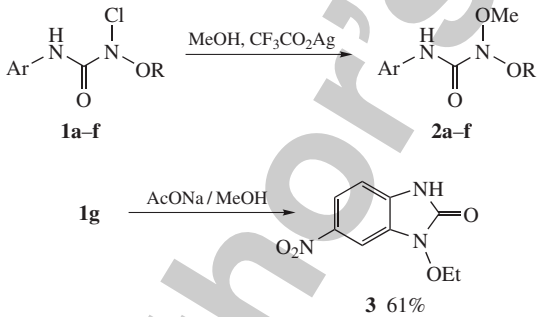
DOI: 10.1016/j.mencom.2011.01.021

Methanolysis of *N*-chloro-*N*-alkoxy-*N′*-arylureas in the presence of silver trifluoroacetate gives the corresponding *N,N*-dialkoxy-*N′*-arylureas, whereas *N*-chloro-*N*-alkoxycarbamates react with alcohols in the presence of silver trifluoroacetate to afford *N,N*-dialkoxycarbamates.

Nucleophilic substitution of the chlorine atom in *N*-chloro-*N*-alkoxyureas depends on the nature of substituent at other nitrogen atom of the urea. *N*-Chloro-*N*-alkoxy-*N′*,*N′*-dimethylureas,[1,2] *N*-chloro-*N*-alkoxy-*N′*-methylureas,[3,4] *N*-chloro-*N*-ethoxy-*N′*-benzylurea[4] and *N*-chloro-*N*-alkoxyureas[5] can be converted into the corresponding *N,N*-dialkoxyureas by their alcoholysis in the presence of base. Such *N*-chloro-*N*-alkoxyureas react similarly with sodium carboxylates to furnish *N*-acyloxy-*N*-alkoxy derivatives.[5] In contrast, *N*-chloro-*N*-alkoxy-*N′*-arylureas **1** in the presence of strong base[6] or sodium acetate[4] undergo cyclization into 1-alkoxybenzimidazol-2-ones. This cyclization may be considered as a result of intermolecular nucleophilic substitution at the nitrogen atom. Thus, the direct conversion of *N*-chloro-*N*-alkoxy-*N′*-arylureas **1** into *N,N*-dialkoxy-*N′*-arylureas **2** looked problematic. The latter seemed to be accessible only by the reaction of *NH*-*N,N*-dialkoxyamines with arylisocyanates.[7]

In the present study we have discovered that *N*-chloro-*N*-alkoxy-*N′*-arylureas **1** can be directly converted to the corresponding *N,N*-dialkoxy-*N′*-arylureas **2** by their methanolysis in the presence of silver trifluoroacetate (Scheme 1).‡



a Ar = 4-$O_2NC_6H_4$, R = Me
b Ar = 4-$O_2NC_6H_4$, R = Bn
c Ar = 4-$O_2NC_6H_4$, R = $Me_2CH(CH_2)_2$
d Ar = 2-$O_2NC_6H_4$, R = Et
e Ar = 4-ClC_6H_4, R = Et
f Ar = 4-BrC_6H_4, R = Et
g Ar = 4-$O_2NC_6H_4$, R = Et

Scheme 1

Replacement of CF_3CO_2Ag by AcONa in the methanolysis resulted in the earlier reported[4] heterocyclization, which was exemplified on transformation of compound **1g** into 1-ethoxy-6-nitro-1,3-dihydro-2*H*-benzimidazol-2-one **3**.§

† Geminal Systems, Part 59. For the previous part, see V. G. Shtamburg, E. A. Klots, M. V. Gerasimenko, O. V. Shishkin, R. I. Zubatuk and R. G. Kostyanovsky, *New J. Chem.*, in press.

‡ N-*Chloro*-N-*benzyloxy*-N′-*(4-nitrophenyl)urea* **1b** was obtained by chlorination of *N*-benzyloxy-*N′*-(4-nitrophenyl)urea with Bu^tOCl according to a described procedure.[4] Yield 84%, yellowish crystals, mp 89–91 °C (decomp.). ^{1}H NMR (300 MHz, $CDCl_3$) δ: 5.10 (s, 2H, $NOCH_2$), 7.44–7.52 (m, 5H, Ph), 7.48 [d, 2H, C(2)H, C(6)H, ^3J 9.0 Hz], 7.87 (br. s, 1H, NH), 8.18 [d, 2H, C(3)H, C(5)H, ^3J 9.0 Hz]. FAB MS, *m/z* (%): 324 $[M + H]^+$ (11), 322 $[M + H]^+$ (28), 91 Bn^+ (100). Found (%): Cl, 10.95. Calc. for $C_{14}H_{12}N_3O_4Cl$ (%): Cl, 11.02.

Compounds **1c**,**d**,**f** were synthesized in a similar manner by chlorination of corresponding *N*-alkoxy-*N′*-arylureas. For their characteristics, see Online Supplementary Materials. *N*-Chloro-*N*-alkoxy-*N′*-arylureas **1a**,[6] **1e**[4] and **1g**[4] were reported earlier.

Synthesis of N,N-*dimethoxy*-N′-*(4-nitrophenyl)urea* **2a** *(general procedure)*. The solution of **1a**[6] (0.099 g, 0.403 mmol) in CH_2Cl_2 (2 ml) was added to a cooled to –27 °C solution of CF_3CO_2Ag (0.107 g, 0.484 mmol) in abs. MeOH (5 ml). AgCl was precipitated. The reaction mixture was warmed to 8 °C for 16 h, then AcONa (0.082 g, 1.00 mmol) was added. After that, MeOH was evaporated *in vacuo*, the residue was extracted with CH_2Cl_2 (15 ml). The CH_2Cl_2 extract was evaporated *in vacuo*, the residue was extracted with benzene (15 ml). The benzene extract was evaporated *in vacuo* to yield 0.091 g (93%) of product **2a**, pale yellow crystals, mp 81–83 °C (benzene–hexane). ^{1}H NMR (300 MHz, $CDCl_3$) δ: 3.97 [s, 6H, $N(OMe)_2$], 7.72 [d, 2H, C(2)H, C(6)H, ^3J 9.3 Hz], 8.18 (br. s, 1H, NH), 8.26 [d, 2H, C(3)H, C(5)H, ^3J 9.3 Hz]. ^{13}C NMR (400 MHz, $CDCl_3$) δ: 62.23 (OMe), 118.75 [C(2), C(6)], 124.84 [C(3), C(5)], 142.25 [C(1)], 143.86 [C(4)], 156.24 [NHC(O)]. FAB MS, *m/z* (%): 242 $[M + H]^+$ (82), 210 $[M + H – MeOH]^+$ (100). Found (%): C, 44.79; H, 4.83; N, 17.25. Calc. for $C_9H_{11}N_3O_5$ (%): C, 44.82; H, 4.60; N, 17.42.

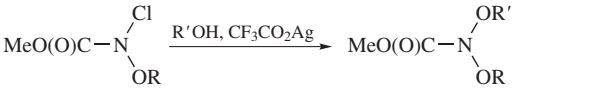
Compounds **2b–f** were synthesized analogously. For their characteristics, see Online Supplementary Materials.

§ *N*-Chloro-*N*-ethoxy-*N′*-(4-nitrophenyl)urea **1g**[4] (0.058 g, 0.223 mmol) was dissolved in solution of AcONa (0.087 g, 0.333 mmol) in MeOH (6 ml) at –30 °C. The solution was heated to 15 °C for 3 h and kept at 15–17 °C for 24 h. Then MeOH was evaporated *in vacuo*, the residue was extracted with CH_2Cl_2 (10 ml), CH_2Cl_2 extract was evaporated *in vacuo*, the residue was crystallized from Me_2CO–CH_2Cl_2, yielding 0.0304 g (61%) of 1-ethoxy-6-nitro-1,3-dihydro-2*H*-benzimidazol-2-one **3**, white crystals, mp 209–211 °C, identified by ^{1}H NMR.[4]

In contrast to *N*-chloro-*N*-alkoxyureas, *N*-chloro-*N*-alkoxycarbamates cannot be converted into *N*,*N*-dialkoxycarbamates by alcoholysis in the presence of bases, which led to reduction products such as *NH*-*N*-alkoxycarbamates and *N*,*N*′-bis(alkoxycarbonyl)-*N*,*N*′-dialkoxyhydrazines.[8,9] However, reaction of *N*-chloro-*N*-alkoxycarbamates with sodium acetate in MeCN gave *N*-acetoxy-*N*-alkoxycarbamates, which can be easily transformed into *N*,*N*-dialkoxycarbamates on alcoholysis.[5]

Here we have found that *N*-chloro-*N*-alkoxycarbamates **4a–c**¶ can be directly converted into the corresponding *N*,*N*-dialkoxycarbamates **5a–d**†† by their alcoholysis in the presence of silver trifluoroacetate (Scheme 2).

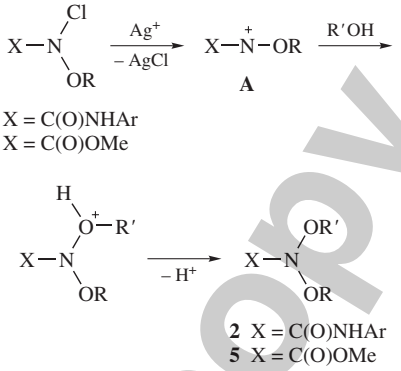
In this manner, sterically hindered *O*-methyl-*N*,*N*-diisopropyloxycarbamate **5c** is obtained with moderate yield by isopropanolysis of compound **4a**. Note that isopropanolysis of *O*-ethyl-*N*-acetoxy-*N*-metoxycarbamate[5] does not lead to *N*,*N*-dialkoxycarbamate.



4a R = Pr^i
4b R = Et
4c R = C_8H_{17}

5a R = Pr^i, R′ = Me, 58%
5b R = Pr^i, R′ = Et, 59% (from **4a**), 45% (from **4b**)
5c R = R′ = Pr^i, 59%
5d R = C_8H_{17}, R′ = Me, 76%

Scheme 2



Scheme 3

Earlier Glover[10–12] and Kikugawa[13,14] proposed the general method for the synthesis of O,N-containing heterocycles by intramolecular cyclization of *N*-chloro-*N*-alkoxycarboxamides in the presence of silver salts, which proceeded *via* generation of *N*-alkoxynitreniun ions.[10–14] Probably, in our case formation of *N*,*N*-dialkoxy-*N*′-arylureas **2** and *N*,*N*-dialkoxycarbamates **5** also proceeds *via* the step of *N*-alkoxynitreniun ions **A** (Scheme 3).

Previously,[15] the structure of the simplest *N*,*N*-dimethoxyurea has been studied. Herein, the XRD study of *N*,*N*-dimethoxy-*N*′-(4-nitrophenyl)urea **2a** was performed (Figure 1).‡‡ The degree of pyramidalization at amide nitrogen atom N(1) in urea **2a** is high enough, as the sum of bond angles centered on this nitrogen atom is 324.0(2)°, the deviation of N(1) atom from the plane of bonded atoms is 0.508(3) Å. This degree of pyramidalization is close to those of *N*-acyloxy-*N*-alkoxybenzamides[16] and *N*-acyloxy-*N*-alkoxyureas.[15]

The N(1)–C(1) amide bond is much longer [1.441(3) Å] compared to the N(2)–C(1) amide bond [1.357(3) Å] owing to the higher conjugation of the planar N(2) atom with carbonyl group.

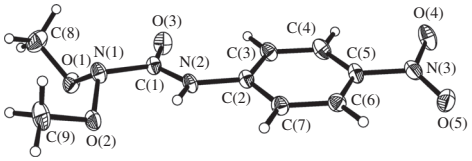


Figure 1 Crystal structure of *N*,*N*-dimethoxy-*N*′-(4-nitrophenyl)urea **2a**. Selected bond lengths (Å) and bond angles (°): O(1)–N(1) 1.418(3), O(2)–N(1) 1.412(3), O(1)–C(8) 1.428(3), O(2)–C(9) 1.437(3), O(3)–C(1) 1.204(3), N(1)–C(1) 1.441(3), N(2)–C(1) 1.357(3), N(2)–C(2) 1.401(3); O(2)–N(1)–O(1) 106.46(19), O(1)–N(1)–C(1) 108.0(2), O(2)–N(1)–C(1) 109.5(2), O(3)–C(1)–N(2) 126.8(2), O(3)–C(1)–N(1) 119.6(2), N(2)–C(1)–N(1) 113.4(2).

¶ *Methyl* N-*chloro*-N-*isopropoxycarbamate* **4a** has been synthesized by chlorination of methyl *N*-isopropoxycarbamate with Bu^{t}OCl by reported procedure,[5] yellowish liquid. ^{1}H NMR (300 MHz, $CDCl_3$) δ: 1.28 (d, 6H, NOCHMe_2, ^3J 6.3 Hz), 3.91 (s, 3H, CO_2Me), 4.31 (sept., 1H, NOCHMe$_2$, ^3J 6.3 Hz). IR (ν/cm^{-1}): 1780 (C=O). Found (%): Cl, 21.04. Calc. for $C_5H_{10}ClNO_3$ (%): Cl, 21.15.

Methyl N-*chloro*-N-*ethoxycarbamate* **4b** has been synthesized by chlorination of methyl *N*-ethoxycarbamate with Bu^{t}OCl by reported procedure,[5] yellowish liquid. ^{1}H NMR (300 MHz, $CDCl_3$) δ: 1.31 (t, 3H, NOCH$_2Me$, ^3J 6.9 Hz), 3.92 (s, 3H, CO_2Me), 4.07 (q, 2H, NOCH_2Me, ^3J 6.9 Hz). IR (ν/cm^{-1}): 1795 (C=O). Found (%): Cl, 22.85. Calc. for $C_4H_8ClNO_3$ (%): Cl, 23.09.

Methyl *N*-chloro-*N*-octyloxycarbamate **4c** was reported earlier.[5]

†† *Synthesis of methyl* N,N-*diisopropoxycarbamate* **5c** (*general procedure*). Methyl *N*-chloro-*N*-isopropoxycarbamate **4a** (0.673 g, 4.017 mmol) was dissolved in Pr^{i}OH (2 ml) at cooling to –27 °C, and the obtained solution was rapidly added to the solution of CF_3CO_2Ag (1.065 g, 4.821 mmol) in Pr^{i}OH (5 ml) at –27 °C, the reaction mixture was warmed to 11 °C for 19 h, then AcONa (0.46 g, 5.61 mmol) was added, the mixture was stirred for 2 h, and the solid formed was filtered off. The filtrate was concentrated *in vacuo*, the residue was twice extracted with the mixture of CH_2Cl_2 (7 ml) and C_6H_{14} (5 ml). The combined extracts were evaporated *in vacuo*. The residue was distilled *in vacuo* to afford 0.456 g (59%) of product **5c**, colourless liquid, n_D^{20} 1.4189. ^{1}H NMR (300 MHz, $CDCl_3$) δ: 1.29 (d, 12H, NOCHMe_2, ^3J 6.3 Hz), 3.85 (s, 3H, CO_2Me), 4.28 (sept., 2H, NOCHMe$_2$, ^3J 6.3 Hz). Found (%): C, 50.31; H, 8.71; N, 7.08. Calc. for $C_8H_{17}NO_4$ (%): C, 50.25; H, 8.96; N, 7.32.

Methyl N-*isopropoxy*-N-*methoxycarbamate* **5a** was obtained similarly to compound **5c** by methanolysis of **4a**, yield 58%, colourless liquid, bp 50–53 °C (3 Torr.), n_D^{23} 1.4168. ^{1}H NMR (300 MHz, $CDCl_3$) δ: 1.30 (d, 6H, NOCHMe_2, ^3J 6.0 Hz), 3.79 (s, 3H, NOMe), 3.87 (s, 3H, CO_2Me), 4.28 (sept., 1H, NOCHMe$_2$, ^3J 6.0 Hz). IR (ν/cm^{-1}): 1770 (C=O). EI MS, m/z (%): 163 M$^+$ (3.4), 105 (5.6), 91 (14.0), 60 (21.3), 59 (54.8), 58 (24.3), 46 (16.9), 45 (36.7), 44 (21.3), 43 (100). Found (%): C, 44.23; H, 8.17; N, 8.42. Calc. for $C_6H_{13}NO_4$ (%): C, 44.17; H, 8.03; N, 8.58.

Methyl N-*ethoxy*-N-*isopropoxycarbamate* **5b** was obtained similarly to compound **5c** by ethanolysis of **4a**, yield 59%, and by isopropanolysis of **4b**, yield 45%, colourless liquid, n_D^{21} 1.4200. ^{1}H NMR (300 MHz, $CDCl_3$) δ: 1.293 (t, 3H, NOCH$_2Me$, ^3J 7.2 Hz), 1.295 (d, 6H, NOCHMe_2, ^3J 6.3 Hz), 3.86 (s, 3H, CO_2Me), 4.06 (q, 2H, NOCH_2Me, ^3J 7.2 Hz), 4.28 (sept., 1H, NOCHMe$_2$, ^3J 6.3 Hz). Found (%): C, 47.19; H, 8.67; N, 7.74. Calc. for $C_7H_{15}NO_4$ (%): C, 47.45; H, 8.53; N, 7.90.

Methyl N-*methoxy*-N-*octyloxycarbamate* **5d**[5] was obtained similarly to compound **5c** by methanolysis of **4c**,[5] yield 75%, and was identified by ^{1}H NMR.

‡‡ *Crystal data for* **2a**. Crystals were grown from CH_2Cl_2–C_6H_{14} at –20 °C, $C_9H_{11}N_3O_5$, M = 241.21, monoclinic, space group $P2_1/c$, at 100 K, a = 4.8371(3), b = 16.9591(11) and c = 12.7986(9) Å, β = 92.609(7)°, V = 1048.82(12) Å^3, F(000) = 504, d_{calc} = 1.528 g cm^{-3}, Z = 4, μ = = 0.126 mm^{-1}. Data were measured using an Xcalibur 3 diffractometer (graphite-monochromated MoKα radiation, $2\theta/\theta$ scan). Selected crystal is found to be a non-merohedral twin due to 180° degree rotation along the a axis with relative contributions of twin components of 0.58 : 0.42. Total 9049 reflections were measured up to $2\theta_{max}$ = 57.74°, of which 4131 are unique (R_{int} = 0.072). The structure was solved by direct method using the SHELX-97 program package.[20] Refinement against F^2 in an anisotropic approximation (the hydrogen atoms isotropic in the riding model) by a full matrix least-squares method for 4037 reflections was carried out to wR_2 = 0.145 [R_1 = 0.059 for 2316 reflections with $F > 4\sigma(F)$, S = 0.98].

CCDC 776941 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre *via* www.ccdc.cam.ac.uk/data_request/cif. For details, see 'Notice to Authors', *Mendeleev Commun.*, Issue 1, 2011.

Such difference between the N–C amide bonds is also observed for unsubstituted *N*,*N*-dimethoxyurea,[15] *N*-chloro-*N*-alkoxyureas[4] and *N*-acyloxy-*N*-alkoxyureas.[15,17] The lengths of the N(1)–O(1) and N(1)–O(2) bonds [1.418(3) and 1.412(3) Å, respectively] are somewhat greater than those of the N–O bonds in *N*,*N*-dimethoxyurea (1.397 and 1.401 Å[15]).

The lone pair (Lp) of the N(1) atom has orthogonal orientation with respect to the amide group plane [the O(3)–C(1)–N(1)–Lp(N1) torsion angle is 83°]. Contrary to *N*,*N*-dimethoxyurea,[15] the both methoxy groups are oriented towards the LpN(1): the C(8)–O(1)–N(1)–Lp(N1) and the C(9)–O(2)–N(1)–Lp(N1) torsion angles are 7° and 33°, respectively. The phenyl group is coplanar to the amide one [the C(3)–C(2)–N(2)–C(1) torsion angle is 5.4(4)°]. The nitro group is slightly turned round the cycle plane [the C(4)–C(5)–N(3)–O(4) torsion angle is 13.4(4)°].

The observed molecule conformation is probably additionally stabilized by weak intramolecular hydrogen bonds N(2)–H(2)···O(2) (H···O 2.13 Å, N–H···O 108°) and C(3)–H(3)···O(3) (H···O 2.24 Å, C–H···O 122°).

In the crystal molecules of *N*,*N*-dimethoxy-*N*′-(4-nitrophenyl)-urea **2a** are linked by intermolecular hydrogen bonds N(2)–H(2)···O(4′) [–1 + *x*, 0.5 – *y*, –0.5 + *z*] (H···O 2.27 Å, N–H···O 155°) and stacking interactions between π-systems of two molecules connected by a translation along the *a* crystal axis [the phenyl ring center lies by 3.50 Å above the center of the C(1)–N(1) bond].

Thus, the nitrenium ions generation from *N*-chloro-*N*-alkoxy-*N*′-arylureas and *N*-chloro-*N*-alkoxycarbamates by the action of silver trifluoroacetate in alcohol media allows for the straightforward access to *N*,*N*-dialkoxy-*N*′-arylureas and *N*,*N*-dialkoxycarbamates, respectively. These kinds of *N*,*N*-dialkoxyamides are known as the starting compounds in the synthesis of valuable *NH*-*N*,*N*-dialkoxyamines.[18,19]

This work was supported by the Russian Academy of Sciences and the Russian Foundation for Basic Research (grant no. 09-03-00537a).

Online Supplementary Materials

Supplementary data associated with this article can be found in the online version at doi:10.1016/j.mencom.2011.01.021.

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Received: 2nd June 2010; Com. 10/3536